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A 19 CM DEBYE-SCHERRER CAMERA FOR WORKING BETWEEN 400°K AND 106°K

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(*Received February* 1, 1964)

ABSTRACT. An adjustable temperature 19 cm Debye-Scherrer camera has been described, in which the specimen can be kept at any steady temperature from 106°K to 400°K, using liquid oxygen as refrigerant and with the help of a small heating element to counterbalance the cooling. Details of construction and performance have been described. The camera is designed for the main purpose of studying thermal expansion coefficients and phase transitions of various substances.

INTRODUCTION

A scheme to investigate the thermal expansion of certain inorganic substances necessitated the construction of an X-ray camera, in which the powdered specimen may be kept at any steady temperature from about 90°K to about 400°K, for several hours at a stretch. Such a camera, using conduction cooling (and heating), has been designed and constructed in the workshop of this Association.

In the adjustable temperature X-ray cameras designed for work in low temperature regions, cooling of the specimen is achieved usually either by the flow of a cooled gas around the specimen, or by conduction. The latter method has been chosen for the present design, because of its easier and better adjustability, and possibility of attaining lower temperature, as well as due to the fact that in the present case of a camera using only powdered specimen, no great constructional complication arises. Also, it will be seen that the same accessories are used for attaining the moderately high range of temperatures necessary for the investigations envisaged.

DESCRIPTION AND OPERATION OF THE CAMERA

The camera may be conveniently described under the two following heads : (1) the film-cylinder and collimator, and (2) the specimen-cooling system, and may be clearly understood with reference to fig. 1.

(1) The film-cylinder (A) is of a strictly uniform outside diameter of 19 cm., with cylindrical slots running almost all round the middle except two diametrically opposite parts, where the collimator (B) and the exit port (C) for the

direct X-ray beam are situated. Two strips of film are wrapped outside the cylinder over the slot, as is usual in standard cameras of similar diameter. The slots, along which the films are to be exposed, end in sharp edges, so that the

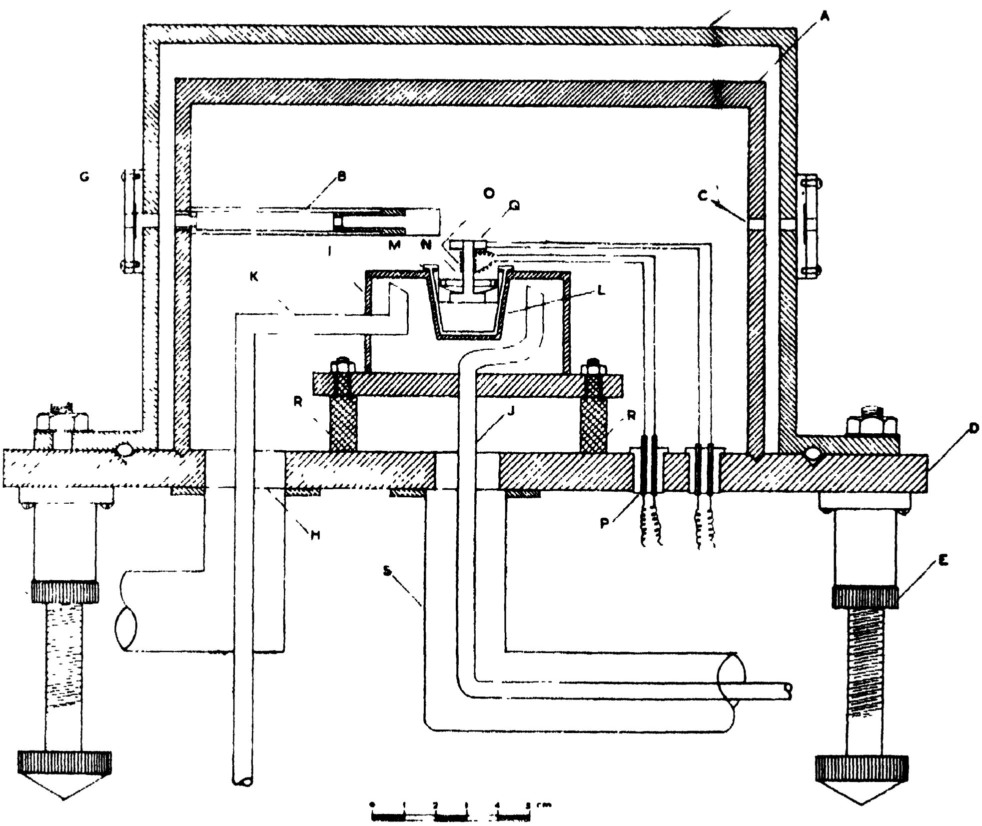

Fig. 1. The 19 cm adjustable temperature Debye-Scherrer camera.

A —film-cylinder
B —collimator
C —exit port for direct beam.
D —base-plate
E —levelling screws
F —air-tight cover
G —aluminium foil window
H —aperture for evacuation
I —liquid oxygen receptacle
J —liquid oxygen inlet tube
K —liquid oxygen outlet tube
L —copper cup
M —copper cone
N —specimen-holder
O —heater
P —sealed exit for leads
Q —thermocouple
R —ebonite pillars
S —stainles-steel vacuum jacket.

exposed parts on the films end in sharp lines, necessary for avoiding film-shrinkage error. Black paper pasted inside the cylinder along the slots and removable phosphor-bronze cover strips with black felt edges outside the cylinder make it a light-tight film carrier. The lower rim of the film-cylinder is chamferred to fit snugly into a circular V-groove on the Duralmin base-plate (D), so that it can be replaced in an identical position on the base plate, every time it is lifted and removed to a darkroom for loading or developing the film. The base-plate is supported on three levelling screws (E).

The collimator (B) is attached to the film-cylinder. It projects wholly inside the cylinder, its axis coinciding with the diameter of the latter, and ends in a taper, so as not to obstruct diffracted rays from reaching the part of the film extending over the entire slotted region. The construction of the collimator is more or less the same as that described by Bradley, Lipson and Petch (1941), the two rectangular slits in this case being 1/16″ and 1/32″ in width.

An air-tight cylindrical brass cover (F), with a flange at the lower rim sealed with an O-ring in a groove on the base-plate, encloses the whole arrangement. The cover is furnished with aluminium foil windows (G) for entrance and exit of X-rays (a lead shutter is provided outside the exit window). The enclosure can be evacuated by a vacuum pump connected to an aperture (H) in the base-plate.

(2) The specimen-cooling arrangement consists of a cylindrical copper receptacle (I) for liquid oxygen. Two tubes (J, K) pass into the receptacle, sealed through the base-plate and running through a high vacuum line for sucking liquid oxgen in and the vapour out respectively. The upper cover of the receptacle has a conical cup (L) of copper attached to it in such a way that the cup projects wholly inside the receptacle. A solid copper cone (M) fits rather closely in the cup, and can be rotated smoothly. The specimen-holder (N) is a copper button fitted into a groove in the cone, with a spring and nut arrangement so that small displacements in all horizontal directions can be given to it. The pressure of the spring holds the button in the displaced position, the lower surface of the same being all the time pressed against the upper surface of the cone. This arrangement permits centering of the specimen mounted at the top of the specimen-holder, as well as ensures thermal conduction between the specimen and the liquid oxygen inside the receptacle.

On the stem of the copper button are wound a few turns of thin Nichrome wire, insulated by thin sheet of mica and Araldite. This serves as a heater element (O), and while the liquid oxygen in the receptacle tends to lower the temperature of the specimen, depending upon the rate of suction of the liquid and the small heat leakage from outside, different steady intermediate temperatures can be obtained by adjusting the current through this heater element. The hollow space inside the receptacle is filled with wire gauze for proper evaporation ofo xygen and distribution of cold.

With no liquid oxygen in the receptacle, the heater is capable of maintaining different steady high temperatures of the specimen up to about 400°K. The connecting leads from the heater are kept free during centering of the specimen, so that the rotation of the cone is not obstructed, and are afterwards connected to the terminals provided at the camera base (P), and passing through polystyrene plugs sealed leak-tight to the base with Araldite. For measurement of temperature, a ring-shaped junction of a calibrated copper-constantan thermocouple (Q) is slipped on the stem of the specimen holder, just below the speimen, after centering has been secured. The e.m.f. of the couple is read off on a calibrated 6″ dial millivoltmeter, each small division of which corresponds to about 2°K in the medium and high temperature ranges.

The liquid oxygen receptacle is mounted on three ebonite pillars (R) on the base-plate with suitable nuts and bolts. During this mounting, the rotation axis of the copper cone was made to coincide with the axis of the film-cylinder. This was checked optically, and once this arrangement was reliably ensured, centering of specimens reduced to the easy task of making them coincide with the axis of rotation of the copper cone.

The liquid oxygen inlet tube (J) is connected to the bottom centre of the receptacle. Inside the chamber, it reaches almost up to the upper cover. The exit tube (K) is attached to the side of the receptacle, and also reaches the same height inside. A stainless-steel jacket (S), in which vacuum is maintained along with that in the camera enclosure during operation, surrounds the whole length of the inlet tube from the camera-base to the liquid oxygen reservoir.

The liquid oxygen reservoir (not shown in the diagram) consists of a cylindrical container of German silver, placed in a 4-pint thermoflask with cotton linings. The container has four openings at the top. Through one of these, the vacuum-jacketted inlet tube enters the reservoir. The jacket fits closely at the entrance, and the inner tube reaches nearly the bottom of the container. A second opening is sealed with a graduated glass tube, closed at the top. The long, thin stem of a hollow glass float projects inside this tube, its position indicating the level of liquid oxygen in the reservoir. The third opening is used for pouring liquid oxygen in the container and is kept closed with a tight fitting cork during operation. The fourth opening connects the upper space over liquid oxygen level of the container to the atmosphere through a series of calcium chloride towers, so that when liquid oxygen is sucked out, only dry air can enter the reservoir.

A suction pump is connected to the free end of the exit tube outside the camera. With the specimen centered, the thermocouple ring placed in position, the connections to the heater secured, and the loaded film-cylinder replaced, the camera chamber is evacuated, and the suction pump started. The rate of suction is controlled by adjusting an air-leak in the pumping system. Liquid oxygen from the reservoir travels through the vacuum-jacketted tube towards the receptacle, and after the initial cooling off, tends to maintain a steady temperature which depends on the rate of heat leakage by radiation, and is finally balanced nicely by adjusting the heater current from a large 6 volt accumulator. Increasing the rate of flow of liquid oxygen and diminishing the heater current, successively lower temperatures are attained. For reaching the lowest temperature the heater is altogether cut out and oxygen allowed to accumulate in the receptacle. Owing to rather large radiation leakage and temperature gradient from the receptacle to the specimen, the lowest temperature reached was about 106°K.

During the many trial operations it has been found that the different temperatures of the specimen can be maintained satisfactorily steady for several hours over which an X-ray exposure is given, the maximum fluctuation never exceeding, and generally being less than .5°K. Five litres of liquid oxygen are found to be sufficient for 7 hours exposure of a specimen maintained at 106°K (not taking account of the period necessary to bring down the temperature of the specimen initially).

ACKNOWLEDGMENT

The author expresses his deep sense of gratitude towards Prof. A. Bose, D.Sc., F.N.I., for his kind interest and encouraging guidance throughout the course of the work. His sincerest thanks are also due to Mr. A. K. Sarkar, Superintendent of the Association's Workshop, who with his team of excellent mechanics, carried out the actual constructions with all possible care.

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